SYNTHESIS OF STACKED GRAPHENE SHEETS BY A SUPERSONIC THERMAL PLASMA TECNHNIQUE AND THE AFFECT OF SAMPLE COLLECTION CHAMBER PRESSURE

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There is active search for graphitized nanostructured carbon with typically higher electrical conductivity combined with large specific surface area, to be used as a catalyst in Fuel Cell applications or as a catalyst support [1]. We are using a segmented plasma torch produced supersonic plasma jet assisted experimental reactor system for synthesis of these nanomaterials, which efficiently combines advantages of larger residence time in the expanded high temperature plasma jet along with processing in a low pressure less impurity prone environment, in addition to the typically high throughput of thermal plasmas [2-4].

Acetylene which is used as the seed material is injected into the core of the plasma just ahead of the torch anode, but upstream of a converging nozzle through which the jet expands into the sample collection chamber below maintained at low pressure. The typical experimental conditions were plasma current 250 ampere, power 16 kW, plasma argon 20 lpm, acetylene 4.2 lpm and sample collection chamber pressure about 10 mbar. During the experiment acetylene is decomposed to rapidly produce carbon particles which deposit on a substrate kept at 120 mm from the nozzle end, from which they are collected for characterization. X-ray diffraction (XRD, Philips PW 1710, using Cu $K\alpha$ radiation) High Resolution Transmission Electron Microscopy (HRTEM, JEOL JEM 2100, CSMRI, Bhavnagar) and BET (N_2 adsorption at 77 K, CSMRI, Bhavnagar) were used for the characterization of the product materials. The control parameters explored were plasma discharge current, location of acetylene injection and the pressure in the sample collection chamber.

TEM photograph shows typically agglomerated nanoparticles of carbon of around 20 nanometer average sizes. XRD measures the interlayer distance (d_{002}) of the synthesized carbon to be 3.47 $\mathring{A}$ (Graphite is 3.36 $\mathring{A}$) and from BET specific surface area is measured to be 360 m^2/g. Most possibly this is the best combination of both graphitization and large surface area by a plasma assisted method so far [5-7]. Graphitization was seen to improve up to 3.42 for higher current. The comparatively longer residence time in the expanded plasma beam is considered to be responsible for enhanced crystallinity. The crystallinity was seen to improve also with pressure and as the gas was injected at a location closer to the anode

The most significant observation was, the average number of graphene sheets stacked together in a single layer decreases with decreasing pressure in the sample collection chamber (Figure 1 and 2).

This explains superior specific surface area of our carbon nanomaterials synthesized at around 10 mbar pressure as reported above; as highly exfoliated graphene sheets (only 3-4 sheets stacked) at lowest possible pressure has bigger total available surface area. It may be concluded that decreasing pressure further may enable synthesis of singe graphene sheet by this plasma assisted method. HRTEM also reveals (Figure 3) that the graphene sheets get better crystalline in presence of iron particles nucleated along with carbon, while co-injecting Ferrocene vapor into the plasma along with the hydrocarbon gas.

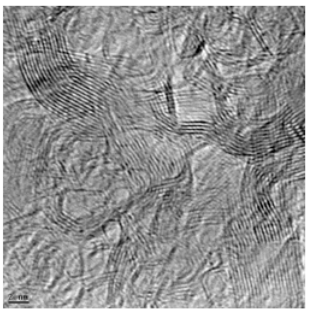

Figure 1: HRTEM photograph of graphene sheets stacked together in single layers, synthesized at 300 mbar sample collection chamber pressure.

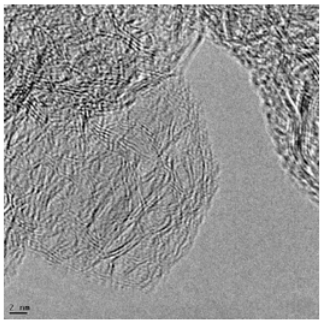

Figure 2: HRTEM photograph of graphene sheets, synthesized at 10 mbar chamber pressure.

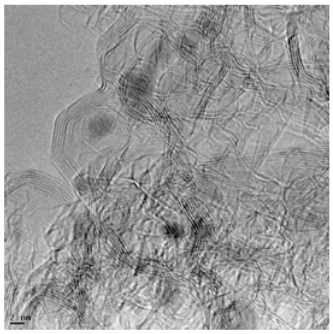

Figure 3: HRTEM photograph of graphene sheets in presence of iron nanoparticles.

References

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